**ABSTRACT**

A versatile electrochemical synthetic route is proposed for the preparation of [Cu2(C8H4O4)4]n metal–organic frameworks. The synthesized composites are characterized by using XRD, SEM, FTIR, and Brunauer–Emmett–Teller (BET) surface analysis. The average particle size was measured to be 8.27 nm and the pore size determined to be 14.06 nm. Here, for the first time, we demonstrate the Cu‐based metal–organic frameworks [Cu2(C8H4O4)4]n as a new class of porous crystalline materials that have the ability to reversibly store Li+ ions. Galvanostatic charge/discharge studies suggest that the terephthalate network reversibly reacts with Li and shows high capacity retention (≈84 % over 50 cycles). The best reversible capacity of 227 mAh g−1 (approximately 95 % of the theoretical capacity) has been achieved in the first cycle at a current density of 24 mA g−1. An easily scalable electrochemical synthesis of the [Cu2(C8H4O4)4]n metal–organic frameworks is an attractive candidate for use with lithium‐ion batteries.